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TECHNICAL MEMORANDUM 1295

THE HEAT OF FORMATION
OF
HEXAUREA ALUMINUM III PERCHLORATE

CHARLES LENCHITZ
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JUNE 1963

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PICATINNY ARSENAL
DOVER, NEW JERSEY

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Picatinny Arsenal Technical Memorandum No. 1295

THE HEAT OF FORMATION OF HEXAUREA ALUMINUM III PERCHLORATE

by

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June 1963

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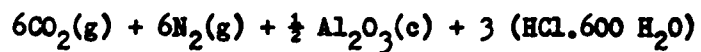
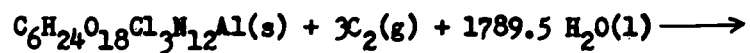
Propellants Laboratory
Picatinny Arsenal
Dover, New Jersey

TABLE OF CONTENTS

	<u>Page</u>
Abstract	1
Introduction	2
Results	
A. Calibration Experiments	2-4
B. Combustion Experiments	4-6
Discussions of Results	7
Experimental	8
References	9
Table I	
Table II	
Distribution List	

ABSTRACT

Hexaurea Aluminum III perchlorate burns smoothly in a combustion bomb leaving an ∞ Al_2O_3 residue. Calculation of the heat of formation from the reaction:



gives a value of -691.53 kcal/mole (at constant pressure and 25°C).

INTRODUCTION

Exploratory combustion experiments with hexaurea Aluminum III perchlorate* indicated that the compound burns smoothly, and leaves a white crystalline residue. An x-ray analysis of this residue showed that only α Al_2O_3 (corundum) was formed**.

Although it was planned to circumvent some of the corrections needed for this type of compound by running comparison experiments it was decided to combust the sample separately because only four 1.2g pellets remained. (A subsequent sample sent to these laboratories caked and turned grey indicating that the aluminum may have separated from the complex).

RESULTS

A. Calibration Experiments

The calorimeter was calibrated with Parr Instrument Company benzoic acid (calorific value 6318 cal/g). The bomb was charged with 35cc of As_2O_3 solution containing the same quantity of HCl as is produced in the sample combustion. A weighed quantity of glass wool saturated with this solution was placed above the sample cup. The benzoic acid was ignited with platinum wire and the rotation was started after the first resistance reading was taken.

Because of these bomb conditions a Washburn correction had to be included for the combustion of benzoic acid. A total of nine corrections were therefore made for each of the five calibration experiments. These corrections are listed in Table 1 and briefly described below.

Step (1) is a correction for the compression of 35cc of solution to 30 atmospheres i.e.

$$\left[\frac{\partial E}{\partial P} \right]_1^{30}$$

The solution was treated as pure water.

* This compound was supplied by Allied Chemical, General Chemical Division, Morristown, N.J.

** X-ray analysis performed by J. Campisi, Propellant Research Section.

Step 2 - Correction for the compression of benzoic acid i.e.

$$\left[\frac{\partial E}{\partial P} \right]_1^{30}$$

Step 3 - Correction for the solubility of oxygen in the solution.
The solution again being treated as pure H₂O i.e.

$$[\Delta E_{\text{soln}}]$$

Step 4 - Correction for the compression of oxygen i.e.

$$\left[\left(\frac{\partial E}{\partial P} \right)_T \right]_0^{30}$$

Step 5 - Correction for the dilution of the HCl solution with water
formed by the reaction.

$$[\Delta E_{\text{diln HCl}}]$$

Step 6 - Correction for the dissolved CO₂ in water and its expansion
to unit fugacity.

$$[\Delta E_{\text{soln CO}_2}]$$

The solution is assumed to
be pure water.

Step 7 - Correction for dissolved oxygen in water $[\Delta E_{\text{soln O}_2}]$
and its expansion to unit fugacity.

Step 8 - Correction for expansion of the gas phase to unit fugacity.

$$[\Delta E_{\text{gas}}]_{P_{\text{final}}}^0$$

Step 9 - Correction for the decompression of the aqueous phase.

$$\left[\frac{\partial E}{\partial P} \right]_{P_{\text{final}}}^0$$

A detailed description of the standardization of calorimeters and the required corrections is described by Neugebauer Ref (1) and by Hubbard, Scott, and Waddington and Prosen Ref (2). The constants used for making these corrections were taken from Ref (2). It should be noted that some corrections are not included because they were deemed insignificant.

B. Combustion Experiments

Combustion of the sample was made in the same environment as in the calibration experiments. Corrections are more complicated than in a routine halogen compound because of the presence of aluminum and nitrogen. 35cc of As₂O₃ solution was used. A similar quantity of glass wool was saturated with this solution (as in the calibration experiments) and the remaining solution placed in the bottom of the bomb. The corrections which were made are shown in Table 2 and are explained below.

Step 1 - Correction for the vaporization of water before firing.

$$[\Delta E_{\text{vap}}]$$

Step 2 - Correction for compression of the 35cc of solution treated as pure water.

$$\left[\frac{\partial E}{\partial P} \right]_T^{30}$$

Step 3 - Correction for the compressibility of the substance was not made. The magnitude of this correction is not significant (in these experiments).

Step 4 - Correction for the solubility of O₂ and N₂ in the solution (treated as pure H₂O). $[\Delta E_{soln}]$

Step 5 - Correction for the compression of the oxygen i.e.

$$\left[\left(\frac{\partial E}{\partial P} \right)_T \right]_{30}^0$$

Step 6 - Correction for the solution of CO₂ in the solution (treated as pure water) i.e. $[\Delta E_{soln}]$

Step 7 - Correction for the solubility of O₂ and N₂ in solution (treated as pure H₂O).

Step 8 - Correction for decompression of the liquid phase.

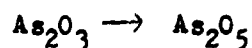
$$\left[\left(\frac{\partial E}{\partial P} \right)_T \right]_{30}^1$$

Step 9 - Correction for nitric acid. Aliquots of each of the four bombs washings were mixed and the nitric acid content was analyzed by the Devarda Method References (4).

Step 10 - Correction for the dilution of HCl to 600 H₂O.

$$[\Delta E_{\text{diln HCl}}]$$

Step 11 - Correction for the reduction of Cl₂ to HCl via



Step 12 - Correction for decompression of gaseous phase i.e.

$$\left[\left(\frac{\partial E}{\partial P} \right)_T \right]_{P_{\text{final}}}^0$$

Step 13 - Correction for condensation of water vapor to liquid i.e.

$$[\Delta E_{\text{vap}}]$$

If the rotation of the bomb is not started at the mid time of the calorimetric experiment and continued through the end of the final rating period a correction for rotation should be included. In the combustion experiments the rotation was started, (approximately), 63 seconds before the mid time. In the calibration experiments the comparative period is 62 seconds. The estimated heat input from bomb rotation, during this period (62-63 seconds) is less than 2 calories. If the correction is therefore omitted from both the calibration and combustion experiments it will not affect the results significantly.

DISCUSSION OF RESULTS

No attempt will be made to estimate the accuracy of the calculated heat of formation because the recovered chloride in the wash water varies from 92.2% to a maximum of only 96.3% of the calculated value. In one determination where this laboratory found 0.509 g chloride an independent check by the Analytical Section yielded 0.497 g. The Volhard Method was used in both cases. Bubbling the gases of combustion through a NaOH solution showed no chloride indicating that none was lost in the exhaustion process. Washing the α Al_2O_3 with hot water also showed the absence of chloride. Based on this evidence it must be concluded that the sample purity is questionable and that the result obtained must be used as an approximate value.

It should be noted that in the two determinations where the gases were analyzed for CO_2 , 98% of the carbon was accounted for. Add to this the solubility of this gas in 35cc of solution remaining in the bomb and one can anticipate a recovery approximating the calculated value.

One additional observation is worthy of note and that is the final form of the Al_2O_3 . Although x-ray analysis showed only α crystals one cannot assume that the amorphous form is completely absent. This assumption seems to be made by Snyder and Seltz in their work on the heat of formation of Al_2O_3 Reference (3). No attempt was made to quantitatively recover the Al_2O_3 .

EXPERIMENTAL

Equipment

The heat of combustion was measured in a rotating bomb calorimeter. The bomb was made by the Parr Instrument Company, Moline, Illinois, and is platinum lined. The calorimeter is a submarine type manufactured by the Precision Scientific Company for accomodating a stationary bomb. A new bucket was therefore designed which accepts the rotating bomb and fits into the calorimeter bath.

The design of the bucket and rotating mechanism is based on prints obtained from the U.S. Bureau of Mines at Bartlesville, Oklahoma.

Resistance measurements were made with a platinum resistance thermometer and a G-2 Mueller Bridge each of which was purchased from the Leeds and Northrup Company, Philadelphia, Pa.

The method used is standard and is adequately described in Reference 2.

Sample

The sample was obtained from Allied Chemical Corp in Morristown, N.J. and combusted as received. Their chemical analysis of the sample is as follows:

	<u>Found</u>	<u>Calculated</u>
Cl	15.5	15.5
Al	3.7 - 3.8	3.935
H	3.99	3.528
C	10.75	10.51
N	---	24.51

A subsequent sample received from Allied Chemical Corporation showed a N content of 24.32 - 24.42%.

REFERENCES

1. Neugebauer, C.A.; "Standard Heats of Formation by Rotating and Stationary Bomb Calorimetry", Thesis Submitted for Ph D (Chemistry) University of Wisconsin 1957.
2. a. Rossini, F.D. "Experimental Thermochemistry", Interscience Publishers, Inc., N.Y. Vol I 1956.
b. Skinner, H.A. "Ibid, Vol. II 1962.
3. Snyder, P.E. and Seltz, H. "The Heat of Formation of Aluminum Oxide", Journal American Chemical Soc. 67,683 (1945).
4. Reiman, Neuss, Naiman, "Quantitative Analysis "International Chemical Series, McGraw Hill Book Co., N.Y. 1942.

Calibration Data
TABLE 1

Run No.	1	2	3	4	5
Wt. (vac)	.997635	1.039035	1.061435	.972285	1.067680
ΔT (corr)	.1581027	.1656443	.1683906	.1548271	.1699340
HNO ₃ (corr)	-1.48	-1.72	-1.48	-1.35	-1.72
Std States	1.89	1.89	1.89	1.89	1.89
Corr	.08	.08	.09	.08	.09
	3.93	3.93	3.93	3.93	3.93
	17.98	17.98	17.98	17.98	17.98
	-2.16	2.25	2.29	2.10	2.31
	-17.20	17.89	18.26	16.78	18.58
	-3.32	3.29	3.27	3.33	3.27
	-21.66	21.85	21.94	21.56	21.96
	-2.11	-2.11	2.11	2.11	2.11
	-22.57	-23.51	-23.98	-22.00	-24.34
Total Cals.	40,019	39,783	39,976	39,828	39,849
Water Equivalent					
Cals/Ohm					
					39,891 \pm 45

Sample Data

TABLE 2

Run No.	1	2	3	4
WT (vac)	1.2256406	1.2093636	1.1955906	1.2161406
ΔT (corr)	.04258226	.04116624	.04068581	.04122991
CO ₂ calculated	---	---	.46015	.46807
CO ₂ recovered	---	---	.45150	.45730
Cl calculated	---	.18750	.18536	.18855
Cl recovered	---	.17724	.17086	.18149
% CO ₂ recovered	---	---	98.1	97.7
% Cl recovered	---	94.5	92.2	96.3
Standard States	4.26	4.26	4.26	4.26
(corr)	-1.89	-1.89	-1.89	-1.89
Cals	---	---	---	---
(1)	-3.93	-3.93	-3.93	-3.93
(2)	-17.98	-17.98	-17.98	-17.98
(3)	3.38	3.33	3.30	3.35
(4)	4.07	4.07	4.07	4.07
(5)	2.02	2.02	2.02	2.02
(6)	None found			
(7)	---	---	---	---
(8)	-1.16	-1.15	-1.14	-1.15
(9)	35.09	31.18	21.06	32.39
(10)	20.45	20.53	20.50	20.56
(11)	-4.34	-4.34	-4.34	-4.34
(12)	40.91	37.10	26.93	38.36
(13)	-1698.65	-1642.16	-1623.00	-1644.70
Total (corr) Cals	-1352.55	-1327.19	-1334.96	-1320.85
$\Delta T \times 39,891$	-927.4	-910.0	-915.3	-905.7
Heat liberated cal/g				
Heat liberated kcal/mole				
ΔH_c (corr to std pressure) kcal/mole =				
				-1333.9 \pm 6.7
				-914.6
				-909.3

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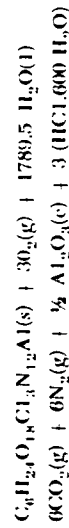
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gives a value of -691.53 kcal/mole (at constant pressure and 25°C).

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Combustion
Bomb
Aluminum oxide

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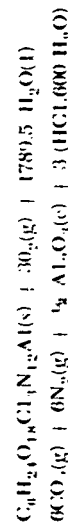
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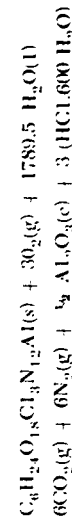
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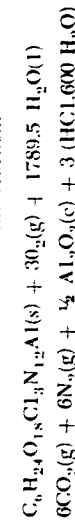
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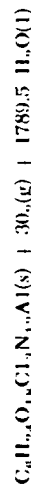
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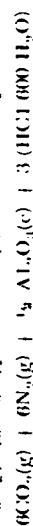
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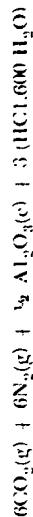
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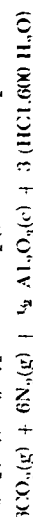
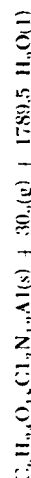
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